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Determination of concentration and diffusion coefficient by the combination of regular and micro electrodes

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It is difficult to estimate that the concentration of solid species which contain unknown amount of water or salt. Such case also occurs when species is soon oxidized with air, when species is sensitive to moisture, when gas is dissolved with bubbling, and when species is in micellar form or ion-paired form. We suggest here a simple voltammetric technique of determining accurate concentrations which cannot be obtained at preparation of solutions. The advantage of the present method is determination of effective concentrations without knowing the diffusion coefficient *D*. Our technique is to take a ratio of voltammetric peak currents at both a mini- and micro-scale electrode.
Two Pt disks 1.6 and 0.1 mm diameter, of which sizes were accurately evaluated, were applied to voltammetric diffusion-controlled currents to determine concentrations of redox species. Since voltammetric peak currents at a large electrode, $I_p = 0.446(\pi a_1^2)Fnc\,(FDv/RT)^{1/2}$, and limiting currents at a microelectrode, $I_L = 4FncDa_2$, are proportional, respectively, to $D^{1/2}$ and D. The ratio, I_p^2/I_L, does not include diffusion coefficient and the concentration. We applied this method to ferrocenyl derivatives, hemin, hexacyanoferrate(II), dioxygen gas and hydrogen gas. New insights are not only the absolute determination of concentrations but also the sensitive detection of adsorption of electrochemical products which blocked the voltammetric currents.